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Spectral Emissivity at High Temperatures

Annual Report for AFOSR Grant 77-3280

Covering Period Feb. 1, 1979 through Jan. 31, 1980

by

R. E. Taylor, D. P. DeWitt, and P. E. Johnson

April 1980

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Purdue University, West Lafayette, Indiana

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  <u>Progress on the development work on the Spectral Emissometer used in conjunction with the high temperature multiproperty apparatus is described. The emissometer has been demonstrated to yield state-of-the-art accuracy for the spectral emissivity in the 1.5 to 15 micrometer range above 1300K for opaque solids. Both electrically conducting and opaque insulating samples of high technological interest have been studied. The present work was aimed primarily at increasing sensitivity to allow measurements at lower temperatures and to provide the basis for investigating semi-transparent materials.</u>			

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TPRL 216

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A. D. BLODGE  
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### 1. Research Objectives

The overall research objective is to develop a state-of-the-art emissometer capable of measuring spectral normal emissivity of samples of electrically conducting materials, of non-conducting opaque materials and of semi-transparent materials at high temperatures. The successful conclusion of this objective will result in a unique apparatus that will import on high technological applications such as laser hardening materials, carbon/carbon composites, turbine blades, combustor liners and electronic materials processing. The apparatus has been developed to the point where operation on opaque materials, both electrical conductors and insulators, is becoming a routine operation. The various parameters that control the accuracy of the measurements, such as blackbody quality, temperature gradients and veiling glare effect have been studied extensively. Test results have been obtained on tantalum, silicon carbide, silican nitride, carbon/carbon composites and pyroceram at temperatures between 1300 and 3000K which have demonstrated the accuracy and reproducibility of the apparatus above 1300K. The present work has as its objectives improving the sensitivity so that measurements can be made at lower temperatures and developing the basis for measuring semi-transparent materials.

## 2. Status of the Research

### A. Introduction

Reliable data on the high temperature radiative properties of many important technical materials, particularly newer alloys, is lacking. The effects on the material properties due to thermal cycling, environmental interactions, and fabrication methods are known to be important. Without a body of solid experimental data generated under well-characterized conditions, rational prediction of material behavior is not possible. There are many high technology applications which have a very basic need for accurate property data for use in heat transfer computations. High technology applications such as turbine blades, combustor liners, laser hardening systems, and electronic materials processing have stringent requirements for radiative properties of advanced materials, particularly nonconducting ones, at high temperatures approaching their destruction/degradation temperatures in the infrared spectral region. Unfortunately, few laboratories have the capability to provide these property measurements.

The ceramics of silicon carbide and silicon nitride are typical of the new generation of materials. Improvements in their high temperature mechanical and thermal properties have resulted from modified hot-pressing fabrication methods using various dopants and temperature treatments. Pyroceram has generated a great deal of interest because of its unique properties. Graphite composites are another class of materials which, through new fabrication methods, have superior high-strength/high-temperature characteristics. In order to effectively utilize these advanced materials there is a need for base-line data to study their behavior at high temperatures.

The Multiproperty Apparatus is a unique and extremely important device that was developed at the Thermophysical Properties Research Laboratory at Purdue University. It has proven to be a powerful scientific and engineering tool for experimentally determining several thermophysical properties at high temperatures. The emissometer was developed to extend the capabilities of the Multiproperty Apparatus to include measurements of the normal spectral emissivity of conducting and nonconducting materials in the 1.5 to 15.0  $\mu\text{m}$  spectral region. Measurements have been made on samples of the ceramics, silicon carbide and silicon nitride, obtained from eight commercial suppliers. Results for the 1300 to 1900K range are in poor agreement with available literature data. Evidence suggests that impurities (such as boron) and phase ( $\alpha$  vs.  $\beta$ ) have important influences on the magnitude and shape of the emissivity spectra. Particularly important to the user is that large variations (as much as 50%) in the property values can occur from one manufacturer's ceramic to another. Two samples of boron carbide were measured at 1900K in an effort to explain the peak in the silicon carbide spectral emissivity data previously reported. A sample of pyroceram from the Air Force Materials Laboratory (AFML) was measured to determine its temperature dependence at 10.6  $\mu\text{m}$ . Measurements were also made at 3.0 and 5.0  $\mu\text{m}$  for this sample. The feasibility of emissometer operation to 3100K was demonstrated in an investigation to determine the temperature dependence of the spectral emissivity of a graphite composite. Operating experiences at such high temperatures suggests that radiation shielding is desirable to improve temperature uniformity and to reduce electrical power requirements. The feasibility of using the present sample/heating tube arrangement to measure semitransparent materials is presently under investigation.

The research effort for the last nine months has been severely hindered by a problem associated with the preamplifier which magnifies the signal leaving the detector. This is why so few samples were measured during the past year.

### B. Emissometer Improvements

Several improvements were made in the emissometer in order to make it a more accurate measuring instrument. First of all, another infrared detector was purchased. This

detector did not increase the spectral range, but it did increase the sensitivity and consequently, the detectivity. For a given number of photons, this detector produces a larger voltage output than the previously used detector.

All the optical surfaces were cleaned with a lens cleaner and with special lens paper. New KCl and NaCl optical crystal windows were purchased for the belljar. The NaCl prism was repolished by Perkin-Elmer, manufacturers of the model 98 prism monochromator.

A very important technical advancement for the emissometer involved the purchasing of three Oriel optical transmission filters. Filters at 3.0, 5.0, and  $10.6\mu\text{m}$  were purchased. These filters allow frequent checking of the wavelength calibration, thus assuring the accuracy of the data. Eventually, after additional filters are purchased, the filters will replace the polystyrene standard as the sole means of performing the wavelength calibration. Not only are the filters more accurate, they are much simpler to use. This is an extreme advantage considering the importance of the calibration to the successful operation of the emissometer.

A procedures manual was developed which outlines in step-by-step detail the procedures required for making spectral emissivity measurements. It also explains how to perform all operations associated with the emissometer, such as setting up the equipment, putting the sample in position, and performing room temperature resistivity measurements. This document not only serves as a valuable users manual, it will help prevent mistakes by doing things systematically each time measurements are taken.

#### C. Normal Spectral Emissivity of Some Important Ceramics - Discussion of Results

A summary of all the silicon carbide data obtained at 1900K is shown in Fig. 1. It appeared as though the presence of boron/carbon caused a peak in the spectral emissivity data at about  $10.5\mu\text{m}$  followed by a valley at about  $13\mu\text{m}$ . This peak-valley may not be stable at 1900K and above in high vacuum, even though the material was sintered or hot-pressed at significantly higher temperature it probably was not under high-vacuum conditions. The presence of  $\text{Al}_2\text{O}_3$  does not seem to significantly affect the spectral character of the data. In Fig. 1, the General Electric silicon carbide sample is in the  $\beta$ -phase while the three other silicon carbide samples are in the  $\alpha$ -phase. The emissivity of the  $\beta$ -phase material decreased markedly with increasing wavelength in contrast to the  $\alpha$ -SiC. Even in the  $\beta$ -phase material, it was conceivable that the presence of boron/carbon could be noted by the presence of a plateau at about  $11\mu\text{m}$ . To help verify this point, two samples of boron carbide were obtained from Ceradyne. One sample was  $\text{B}_4\text{C}$  and the other was  $\text{B}_4\text{C} + 25\%$  SiC. The normal spectral emissivity for each was obtained at 1900K in an effort to try and explain the "hump" in the silicon carbide data. A comparison of the boron carbide data and the silicon carbide data is shown in Fig. 2. The spectral character of the boron carbide samples is quite unstable from about  $8\mu\text{m}$  on. This data seems to indicate that the cause of the plateau is not due to boron carbide. It is presently suspected that tungsten may be the cause of the peak at about  $10.5\mu\text{m}$ , but further verification of this suspicion is needed.

In any event, it is obvious that the spectral emissivity of silicon carbide can be controlled at the longer wavelengths for particular applications. Since the present silicon carbide samples were diamond ground, they exhibited a polished appearance. The recommended (provisional) curves given by CINDAS for average polished SiC at 1000 and 2400K are also shown in Fig. 1. These values are about 15% below the present values for wavelengths less than  $11\mu\text{m}$  and include the peak-valley found in the present samples.

Another very important set of data was collected for a pyroceram sample supplied by AFML. It was desired to determine the temperature dependence of the pyroceram sample at  $10.6\mu\text{m}$ . In order to obtain highly accurate data, the emissometer was modified. The emissometer was described in great detail in earlier reports and therefore will not be explained here. The emissometer was modified by placing the focusing lens, which is located at the exit plane of the monochromator, and the infrared detector directly behind the chopper. A filter holder that mounted directly on the front of the detector was constructed so that the optical filters could be readily interchanged. With this arrangement, the flux

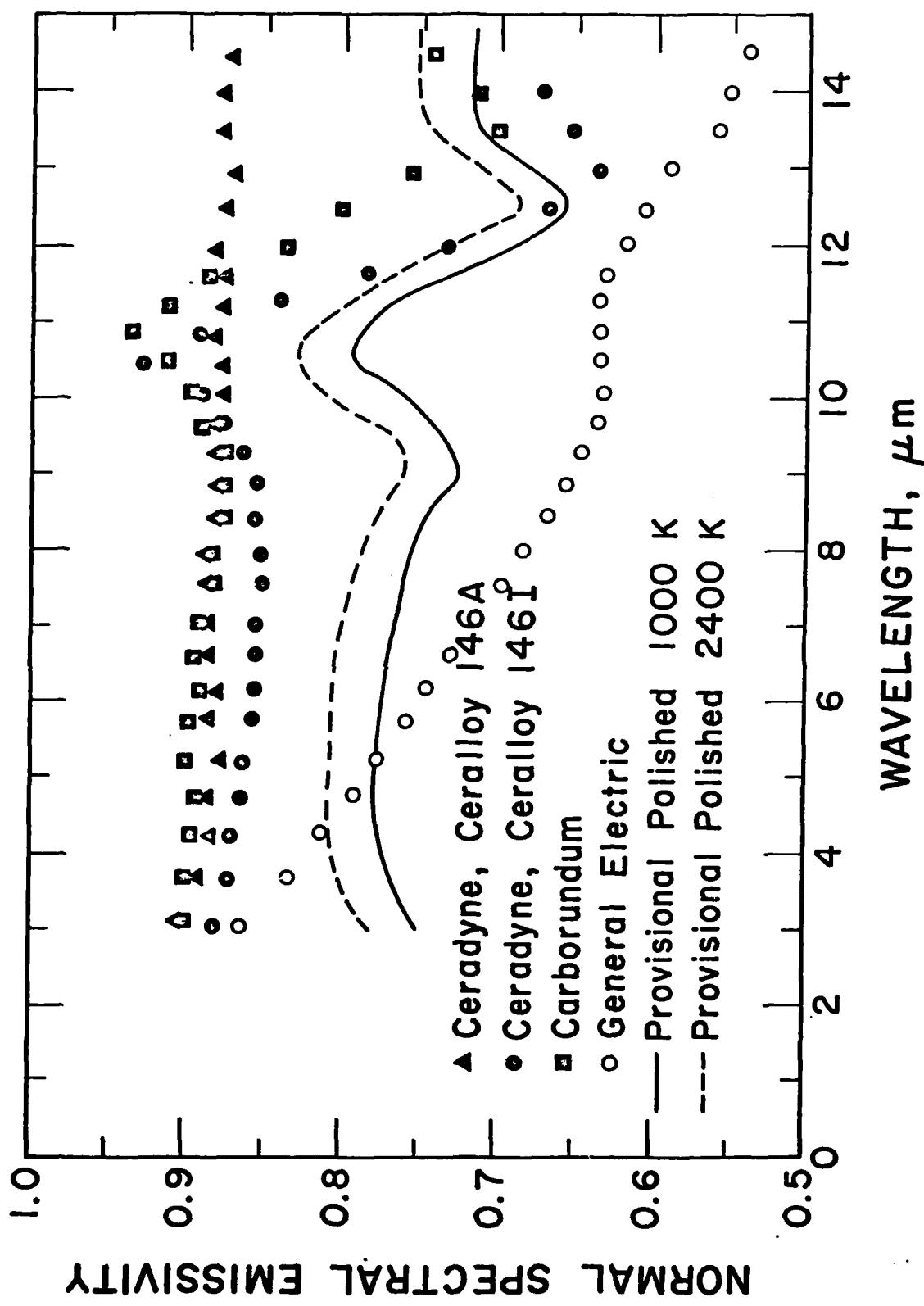


Figure 1. Normal Spectral Emissivity of Silicon Carbide at 1900K.

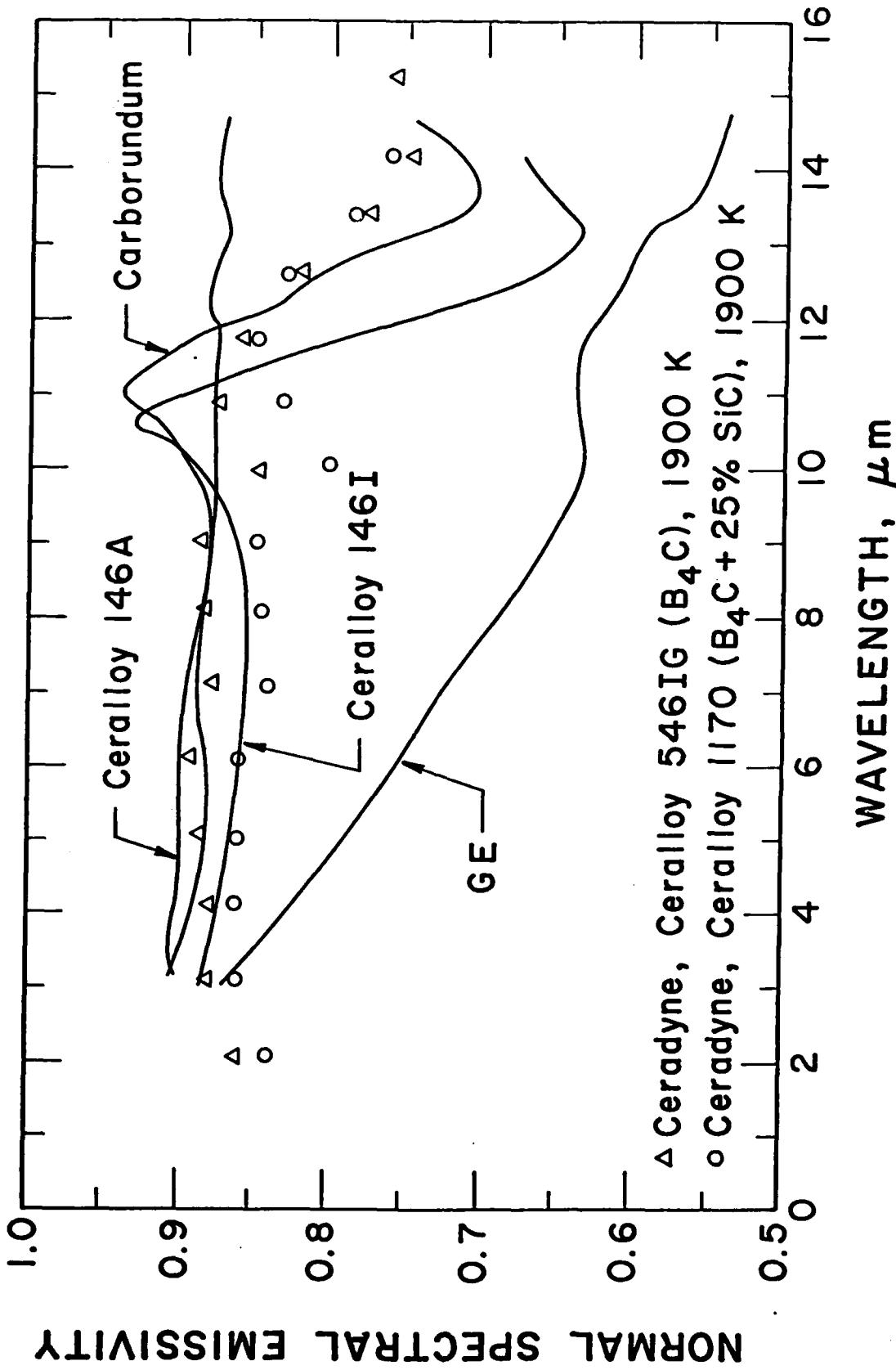


Figure 2. Comparison of the Spectral Emissivity of Silicon Carbide and Boron Carbide at 1900K.

passed through the aperture, through the chopper, through the lens, and then through the optical filter and into the detector. By placing the filter directly in front of the detector window and by eliminating the monochromator from the optical path, very precise data was obtained. Besides making measurements at  $10.6\mu\text{m}$ , data was also collected at  $3.0$  and  $5.0\mu\text{m}$  since these filters were available. The additional data was collected to attempt to verify predicted trends in the pyroceram data. Measurements were made at all three wavelengths from about  $100\text{K}$  to  $1400\text{K}$ . The results are shown in Fig. 3. As can be seen, at each of the wavelengths the curve was rather flat within the temperature range considered. Pyroceram has generated a great deal of interest and many more samples will probably be measured in the near future.

#### D. Present Efforts

Another pyroceram sample will be measured in the immediate future. This sample will be measured from  $1.5$  to  $15.0\mu\text{m}$ . This data, combined with the previous results, should give us a much better understanding of the spectral character of pyroceram at temperatures approaching its degradation temperature.

Within the next week, two samples of Platinum will be measured. This will be a major scientific accomplishment because there is very little spectral data available for Platinum and almost no high temperature data.

Work is presently being done on modeling the semitransparent samples. It is hoped that by mid summer a trial run on a semitransparent material can be performed.

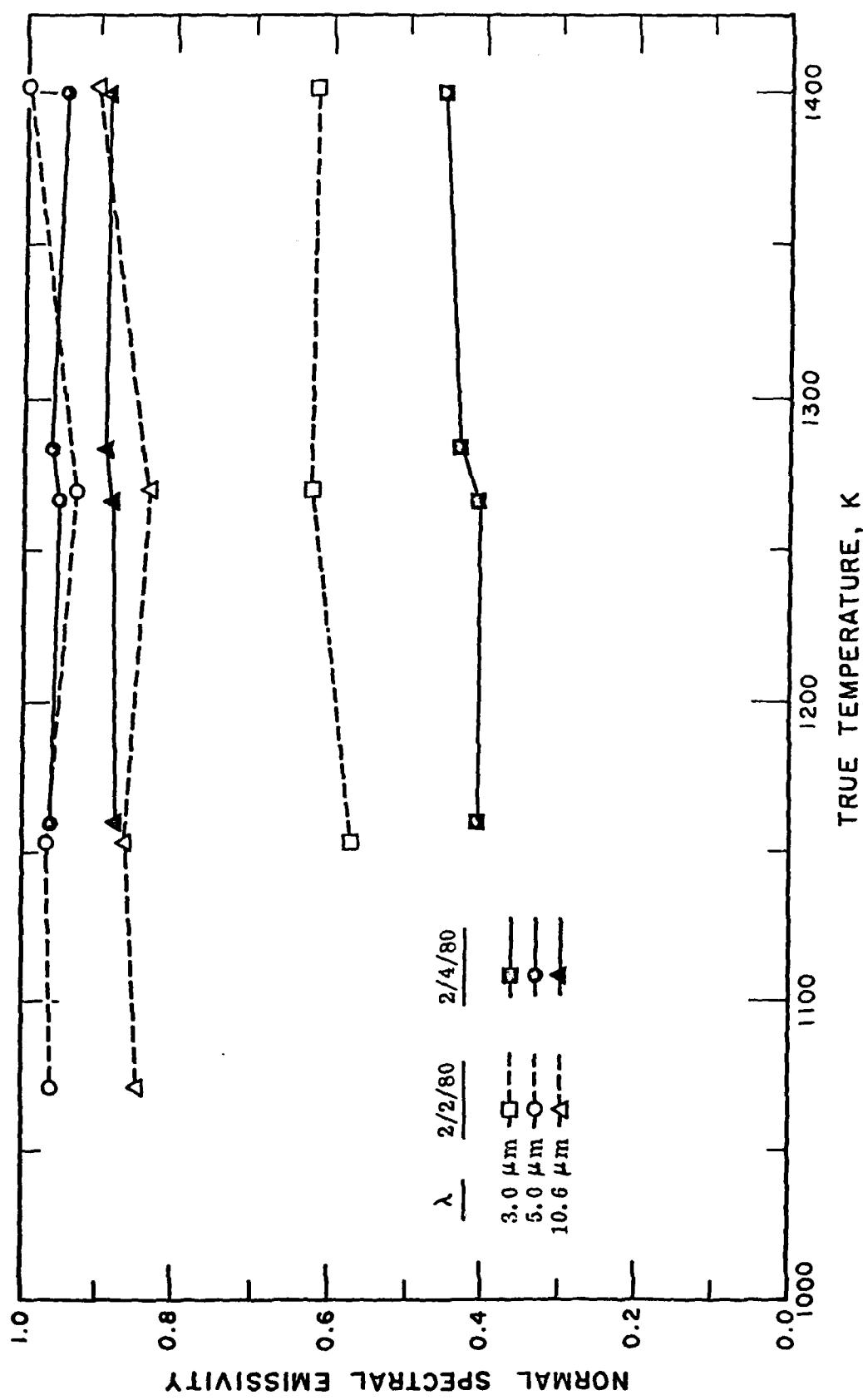


Figure 3. Normal Spectral Emissivity of Pyroceram at 3.0, 5.0, and 10.6 $\mu\text{m}$ .

3. Cumulative Chronological List of Written Publications in Technical Journals

D. P. DeWitt, R. E. Taylor, and T.K. Riddle, "High Temperature Computer Controlled Emissometer for Spectral and Total Measurements on Conducting and Nonconducting Materials," Proceedings Seventh Symposium on Thermophysical Properties, ASME, pp. 256-264, 1977.

P.E. Johnson, D.P. DeWitt, and R.E. Taylor, "Method for Measuring High Temperature Spectral Emissivity of Nonconducting Materials," AIAA, in press, 1980.

R.E. Taylor and H. Groot, "Thermophysical Properties of POCO Graphite," High Temperature-High Pressure, in press, 1980.

4. Technical Reports and Thesis prepared for this Project

- (1) D. P. DeWitt, B. Hoenle, and R. E. Taylor, "Spectral and Total Emissivity and Reflectivity at High Temperature," April 30, 1976, PRL Report 116.
- (2) D. P. DeWitt, R. E. Taylor, and B.D. Hoenle, "Description of the PRL High Temperature Emissometer," November 1976, PRL Report 126.
- (3) R. E. Taylor and D. P. DeWitt, "Spectral and Total Emissivity and Reflectivity at High Temperatures," March 31, 1977, PRL Report 130.
- (4) D. P. DeWitt, T.K. Riddle and R. E. Taylor, "Spectral and Total Emissivity and Reflectivity at High Temperatures," Progress Report to AFOSR, September 15, 1977, PRL Report 136.
- (5) D. P. DeWitt, T. K. Riddle, and R. E. Taylor, "Spectral and Total Emissivity and Reflectivity at High Temperatures," Interim Report to AFOSR, April 23, 1978, AFOSR-TR-78-0901, PRL Report 152.
- (6) R. E. Taylor and H. Groot, "Thermal Properties of POCO Graphite," May, 1978, PRL Report 153.
- (7) P. E. Johnson, D. P. DeWitt, and R. E. Taylor, "Spectral Emissivity at High Temperatures, Six Month's Report to AFOSR," September 1978, AFOSR-TR-78-1480, PRL Report 163.
- (8) T. K. Riddle, "A New Method for Measuring the Spectral Emissivity of Nonconducting Materials at High Temperatures," May 1978, MSME Thesis, School of Mechanical Engineering, Purdue University.
- (9) R. E. Taylor, D. P. DeWitt and P. E. Johnson, "Spectral Emissivity at High Temperatures," Annual report, April 1979, TPRL 189.

### 5. List of the Professional Personnel Associated with the Research Effort

D. P. DeWitt, Professor of Mechanical Engineering, Co-major investigator, responsible for radiometric related procedures and instrumentation.

R. E. Taylor, Senior Researcher, Responsible for multi-properties apparatus, digital data acquisition system and materials science aspects.

T. K. Riddle, Graduate Student, MSME May 1978 Thesis, "A New Method for Measuring the Spectral Emissivity of Nonconducting Materials at High Temperatures."

P. E. Johnson, Graduate Student, MSME May 1980, Thesis, "High Temperature Normal Spectral Emissivity of Several Important Technical Materials," in preparation.

R. L. Shoemaker, Associate Researcher, assisted in computer-related problems.

W. Vaughn, (Research Assistant) assisted in machining operations.

### 6. Interactions

#### A. Spoken papers presented at meetings

- (1) D. P. DeWitt, R. E. Taylor, and T. K. Riddle, "High Temperature Computer Controlled Emissometer for Spectral and Total Measurements on Conducting and Nonconducting Materials," presented at the Seventh Symposium on Thermo-physical Properties, ASME, Gaithersburg, MD, May 10-12, 1977.
- (2) P. E. Johnson, D. P. DeWitt, and R. E. Taylor, "Method for Measuring High Temperature Spectral Emissivity of Nonconducting Materials," presented at the 14th AIAA Thermophysics Conference, June 4-6, 1979, Orlando, Florida.
- (3) D. P. DeWitt, P. E. Johnson and R. E. Taylor, "Spectral Emissivity of Ceramics at High Temperatures: Silicon Carbide and Silicon Nitride," paper 79-HT-24, 18th ASME-AIChE National Heat Transfer Conference, August 5-8, 1979, San Diego, CA.

#### B. Consultive and Advisory Functions

- (1) Mr. Koenig of AFML has sponsored a small grant to measure the spectral emissivity of pyroceram to the softening point. We have measured one sample of this material three wavelengths up through the softening region and will make additional measurements for him.
- (2) We have measured the total emissivity of certain graphitic materials and provided consulting services for the Department of Energy (C. Tarr) for the instrument power system of the Solar Polar Launch Vehicle.
- (3) We are currently measuring the spectral emissivity of several platinum alloys for Owens Corning (K. Bubba).
- (4) We are providing consulting services for Oak Ridge National Laboratory (D. McElroy) in obtaining and installing a multiproperty apparatus for high temperature thermo-physical property research.

**7. Other Statements**

- (a) Dr. A. Ono of the National Research Laboratory of Metrology of Japan has received a government grant to come to this country on a one year's leave. Dr. Ono will spend this time in working on high temperature emissivity of transparent materials in conjunction with Dr. DeWitt on the present grant extension. Dr. Ono will receive no AFOSR funds for his endeavors.
- (b) Dr. R.E. Taylor has been asked by the Commission on Standardization of Methods for Thermophysical Properties Measurements to prepare an extended paper on direct heating methods. This chapter will include the high temperature emissometer developed as part of the AFOSR Grant.
- (c) Dr. R.E. Taylor has been invited to present a lecture at the 7th European Conference on Thermophysics 30 June - 4 July 1980, in Antwerp, Belgium on the subject of direct heating methods. This lecture will include references to the progress made under AFOSR sponsorship.